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SUMMARY

1. Rapid acidity tests employing (a) sodium carbonate, (b) saturated lime, (c) sodium hydroxide and (d) Farrington solutions are described.

2. Precautions to be observed with these tests and the conditions that must be observed in grading cream at 0.2, 0.4 and 0.6 percent acidities with these methods are given.

3. Errors involved through the use of the quart milk bottle as a container in which to prepare the solutions were shown to be unimportant.

4. The concentrations of solutions were found to decrease as a result of exposure to air. This was least with the sodium carbonate and Farrington solutions and greatest with sodium hydroxide. It is advised, therefore, to keep all test solutions tightly stoppered when not in use.

5. The peculiar behavior of the Farrington solution, in that it graded at 0.4 and 0.6 percent, cream samples that had acidities from 0.465 to 0.485 and 0.70 to 0.71 respectively, was shown to result from too great a concentration of alkali and too little phenolphthalein in the tablets.

Rapid Acid Tests for Cream

By E. W. Bird and D. F. Breazeale

The Farrington rapid acid test has been employed for many years by the fluid milk industry for grading samples on the borderline of acidity set for their purchase. In performing the test, neutralizing solutions (containing phenolphthalein) used are of such strength that 1 dipper of solution added to 1 dipper (of the same size) of milk gives a pink mixture if the milk is 0.1 percent acid or less; 2 dippers of neutralizing solution grade at the 0.2 percent point and so on. By adjusting the neutralizing strength the grades corresponding to 1 and 2 dippers of neutralizing solution to 1 of milk can be made 0.2 and 0.4 percent, respectively, instead of 0.1 and 0.2 percent. This is the strength of the solution used for cream grading.

The preliminary experiments conducted with cream indicated that although the Farrington test was satisfactory at 0.2 percent acidity, the use of 2 and 3 dippers of the solution to 1 dipper of cream did not grade the cream at 0.4 and 0.6 percent acid as it should have but actually graded the samples at acidities higher than these points. The rapid acid test, therefore, needs some modification for use with cream.

When the Iowa cream grading law[1] was under consideration the Iowa Agricultural Experiment Station was asked to investigate methods for rapid acid tests for cream so that the creameries and cream stations could, if possible, prepare their own reagents and have definite information regarding the limitations of rapid acid testing methods. This request has been complied with, and the results obtained will be presented as follows:

(a) Directions are available for the performance of rapid acid tests and for the preparation of solutions for use in these tests.

(b.) The experimental data on which certain changes in the standard Farrington procedure and the development of new methods are based are given for the benefit of equipment and reagent manufacturers, control men in cooperative and centralized organizations, state inspectors and field men.

[1] Bulletin 64; Dept. of Agr., State of Iowa, 1935.

DESCRIPTION OF TESTING METHODS

This section of the bulletin is designed chiefly for the operators of the acid test. Directions are given for performing the tests. Dippers of the sizes specified for some of the tests are not now available but the writers understand that they will be made available by supply houses if demand for them arises.

Four methods are satisfactory providing certain precautions are observed. All four methods will be described since from them a test applicable to almost any condition can be chosen. The methods of preparing all testing solutions are described in the Appendix.

SODIUM CARBONATE

In this test the dippers for the cream and neutralizing solutions are of the same size. The cream acidities are determined as follows:

(a.) 0.2 percent. Add 1 dipper of cream and 1 dipper of testing solution to a white cup. Stir thoroughly. If the mixture is pink the cream contains 0.2 percent acidity or less; if colorless, more than 0.2 percent.

(b.) 0.6 percent. Add 1 dipper of cream and 3 dippers of solution to a white cup. Stir thoroughly. A pink color indicates 0.6 percent acidity or less; a colorless mixture, more than 0.6 percent.

(c.) This test is applicable wherever butter moisture scales are available. It can be used where cream scales are available if weights of 2.3 and 0.5 gm. masses are purchased.

(d.) Precautions: 1. Keep solution tightly stoppered when not in use. 2. The anhydrous sodium carbonate which is used to make the solution must be kept well stoppered and dry.

FARRINGTON SOLUTION

(a.) For cream of 0.2 percent acidity: Place 1 dipper of cream in a white cup. With a dipper of exactly the same size add a dipperful of Farrington solution. Mix thoroughly. If the mixture is pink the cream contains 0.2 percent acid or less; if colorless, more than 0.2 percent acid.

(b.) For 0.6 percent cream: To 1 dipper of cream in a white cup add 1 dipper of proper size (larger) of testing

solution and stir. If the mixture is pink the cream is 0.6 percent acid or less, if colorless more than 0.6 percent acid. The dippers used are of different sizes. The relative volumes of cream and solution are such that the solution dipper size is 2.713 times that of the cream dipper.

(c.) This test is applicable to all conditions.

(d.) Precaution: Keep the solution stoppered when not in use.

SATURATED LIME

The cream dipper size is the same at both 0.2 and 0.6 percent acidity with this solution; the solution dipper at the 0.2 level is smaller and at the 0.6 level is larger than the cream dipper.

(a.) For 0.2 percent acidity add 1 dipper of cream to 1 dipper of solution. (The solution dipper size is 1.706 times that the cream dipper.) Add 5 drops of phenolphthalein solution. If it remains pink the cream is 0.2 percent or less acid.

(b.) For 0.6 percent acidity add 1 dipper of cream to 1 dipper of solution. (The solution dipper size is 1.706 times that of the cream dipper.) Add 5 drops of phenolphthalein solution and stir. If the mixture remains pink it is 0.6 percent or less acid.

(c.) This test is adaptable to all conditions but is especially of value to small creameries or to cream stations where costs are particularly important.

(d.) Precautions: 1. Keep solution covered when not in use. 2. Do not stir sediment in dipping lime solution. 3. Do not include scum from top of solution in dipper of solution measured. 4. Use dippers that have correct size ratios. 5. Prepare and keep solutions under conditions specified in Appendix.

SODIUM HYDROXIDE

With this test the cream and solution dippers are again of the same size. The test is performed exactly as was described for sodium carbonate.

This solution is adaptable only in those cases in which a central laboratory can make, standardize and measure for shipment the N/1 sodium hydroxide required.

Precautions: 1. Do not allow solution to stand open to air

when not in use. 2. Exercise care in preparing, measuring, and transferring from shipping container the N/1 sodium hydroxide solution. 3. Do not place indicator in N/1 sodium hydroxide before shipment. This step must be performed at the time of dilution because the strong caustic causes an unnatural action of the indicator.

EXPERIMENTAL WORK ON THE STUDY OF RAPID ACID TESTS

This section of the bulletin is written primarily for the technically trained individual interested in preparing the reagents and equipment for use or for sale, the control chemist, the inspector and the field supervisor.

The following reagents were studied: (1) saturated lime solution, (2) lime solutions prepared by diluting saturated lime, (3) Farrington solution, (4) sodium carbonate solutions and (5) trisodium phosphate solutions. These were studied to determine the following points:

A. The amounts of these reagents required per quart to grade cream at 0.2 percent acidity.

B. The amount of indicator necessary to make the test dependent on the alkali concentration and not on the indicator concentrations.

C. Modifications necessary in the use of the solution that will grade cream at 0.2 percent acidity when used to grade at 0.4 and 0.6 percent acidity.

D. Errors introduced by variation of quart bottle volumes.

E. Errors due to moisture variations in dry reagents.

F. Errors introduced by allowing solutions to remain exposed to the air.

A. and B. **Amounts of Reagents per Quart of Solution.** The preliminary investigations based on the use of 0.15 gm. of phenolphthalein per quart of solution indicated the following concentrations of the various reagents were necessary.

Lime solution:	630 cc. filtered saturated lime solution per quart
Sodium hydroxide:	23.0 cc. N/1 per quart
Sodium carbonate:	2.8 gm. per quart
Trisodium phosphate:	7.8 gm. per quart

It was quickly observed, however, that the alkali concentrations of all these solutions were greater than necessary to neutralize the acid in the cream and that this excess of alkali became apparent when more indicator was added.

For this reason a series of solutions with varying alkali concentrations was prepared. Four solutions were prepared for each concentration having, respectively, 0.15, 0.25, 0.35 and 0.45 gm. phenolphthalein per quart. When these series were run it was found that between 0.35 and 0.45 gm. of phenolphthalein per quart was necessary if the test were not to be a function of the indicator concentration.

It was decided, therefore, that 0.5 gm. phenolphthalein would give a satisfactory excess and at the same time would be an amount which could be weighed conveniently. When solutions were prepared with 0.5 gm. phenolphthalein per quart the following concentrations were found satisfactory for grading at 0.2 percent acidity:

Sodium hydroxide:	21.5 cc. N/1 solution per quart
Sodium carbonate:	2.3 gm. per quart
Trisodium phosphate:	7.4 gm. per quart

It will be noted that the diluted lime solution was not included; it was omitted because on standing it lost phenolphthalein color and a considerable amount of precipitate settled on the bottoms of the bottles. The precipitate was thought to be a slightly soluble calcium salt of the dye. Experimentation with the solution was therefore discontinued.

Although the diluted lime solution containing indicator was not satisfactory, the use of saturated lime solutions with the indicator added to the cream at the time the tests were made was studied because a saturated lime method for acidity grading was published (in 1931) by the Iowa Department of Agriculture.[2] The method recommended, incorporating the indicator in the saturated lime solution, is now deemed inadvisable for reasons stated above. When this method was employed (adding the indicator to the sample of cream but otherwise using the directions recommended),[2] the cream graded as 0.2 percent actually had 0.3 percent acidity and that graded as 0.4 percent had 0.65 percent acidity. From these figures the ratio

[2] Laws and rules relating to dairy products with suggestions on cream grading. Iowa Dept. Agr., p. 20, 1931.

of the sizes of dippers for cream and lime solution for the 0.2 percent point was calculated to be 1 : 0.666.

Saturated lime solution, with these dipper ratios, was checked along with the other solutions for the 0.2 acid point. The results of the tests are shown in table 1.

TABLE 1. THE PRECISION WITH WHICH RAPID ACID TESTS GRADE CREAM AT 0.2 PERCENT ACIDITY.

Reagent	Concentration of solutions	Dipper ratios	Colors when cream acidity was				
		Cream:sol'n.	0.200%	0.207%	0.215%	0.220%	0.235%
Lime water	Saturated solution	1:0.666	pink	(?)*	color-less	color-less	color-less
Sodium hydroxide	21.5 cc. N/1 per quart	1:1	pink	pink	(?)	color-less	color-less
Sodium carbonate	2.3 gm. anhydrous per qt.	1:1	pink	(?)	(?)	(?)	color-less
Trisodium phosphate	7.4 gm. per quart	1:1	pink	(?)	(?)	color-less	color-less
Farrington solution	64 tablets per quart	1:1	pink	(?)	(?)	(?)	color-less

*(?) denotes a pink color fainter than the deeper color on the Nafis stirring rod.

These results indicate that any of the solutions used as indicated in the table are satisfactory and sufficiently precise for grading cream at the 0.2 percent acid level. The results likewise show that the stronger alkalies (lime and sodium hydroxide) give sharper color changes than the buffer salts (sodium carbonate and trisodium phosphate) that were used.

C. **Modifications of above methods necessary if cream is to be graded at 0.4 and 0.6 percent acidities.** When the solutions and dipper sizes given in table 1 were employed to grade cream at the 0.4 and 0.6 percent acidity levels by adding 2 and 3 dippers of neutralizing solution to 1 dipper of cream, the actual acidities of the cream when graded as 0.4 and 0.6 percent by the different solutions are shown in table 2.

These data show that sodium hydroxide and sodium carbonate solutions of the concentrations given may be used in the manner prescribed for the Farrington test, ie., 2 dippers of solution to 1 of cream will grade the cream satisfactorily at 0.4 percent acidity, and 3 dippers of solution to 1 of cream will grade the

TABLE 2. ACTUAL ACIDITIES OF CREAM WHEN GRADED AS 0.4 OR 0.6 PERCENT ACIDITY.

Reagent	Concentration of solutions	Dipper ratios	Actual acidity of cream when graded as	
		Cream:sol'n.	0.4 percent	0.6 percent
Lime water	Saturated solution	1:0.666	between 0.44 and 0.45	between 0.675 and 0.700
Sodium hydroxide	21.5 cc. N/1 per quart	1:1	between 0.40 and 0.415	between 0.60 and 0.61
Sodium carbonate	2.3 gm. anhydrous per qt.	1:1	between 0.40 and 0.415	between 0.61 and 0.635
Trisodium phosphate	7.4 gm. per quart	1:1	between 0.435 and 0.465	between 0.61 and 0.635
Farrington solution	64 tablets per quart	1:1	between 0.465 and 0.485	between 0.70 and 0.71

cream satisfactorily at 0.6 percent acidity, if the dippers employed are the same size. Trisodium phosphate is less satisfactory at the 0.4 percent level, and adjustment of dipper sizes for Farrington and saturated lime solutions is necessary at both the 0.4 and 0.6 levels.

Dipper sizes were calculated for the 0.4 and 0.6 percent points and were adjusted in size experimentally until it was considered that they were correct. The results obtained are presented in table 3.

At first observation the results obtained with sodium carbonate and Farrington solution seem to be at variance since Far-

TABLE 3. THE PRECISION WITH WHICH THE RAPID ACID TESTS GRADED CREAM AT 0.4 AND 0.6 PERCENT ACID.

Reagent	Concentration of solution	0.4 percent cream			0.6 percent cream		
		Dipper ratio cream:sol'n.	cream 0.4	cream 0.43	Dipper ratio cream:sol'n.	cream 0.60	cream 0.62
Lime water	Saturated solution	1:1.241	pink	colorless	1:1.706	pink	colorless
Sodium hydroxide	21.5 cc. N/1 per quart	1:1*	pink	colorless	1:1†	pink	colorless
Sodium carbonate	2.3 gm. per quart	1:1*	pink	colorless	1:1†	pink	colorless
Trisodium phosphate	7.4 gm. per quart	1:1*	pink	colorless	1:1†	pink	colorless
Farrington solution	64 tablets per quart	1:1.862	pink	(?)	1:2.713	pink	(?)

*Two dippers of solution used.
†Three dippers of solution used.

rington tablets are prepared from alkali carbonates. It will be recalled, however, that when the phenolphthalein was increased in the sodium carbonate solutions from 0.15 to 0.45 gm. per quart, the amount of reagent required to grade at 0.2 percent acid was decreased from 2.8 to 2.3 gm. With the Farrington tablets employed it was found that the presence of color in the cream-solution mixture was not wholly a function of the alkali, since, apparently, too little indicator was used in their manufacture. When the Farrington solution is used there is three times as much indicator at the 0.6 percent acid level in twice the volume of solution that there is at the 0.2 percent acid level. There is also 3 times the excess of alkali present at 0.6 as at 0.2 percent acidity. It is felt that this combination of factors explains the discrepancy. This is not the case with the sodium carbonate solution since there is neither excess alkali nor deficiency of indicator at any time.

From the foregoing data it is considered that table 4 presents reliable information concerning the use and preparation

TABLE 4. CONCENTRATIONS OF SOLUTIONS AND RATIOS OF CREAM AND TEST SOLUTION VOLUMES FOR GRADING CREAM AT 0.2, 0.4 AND 0.6 PERCENT ACIDITIES.

Reagent	Concentration of solutions	0.2 percent acidity		0.4 percent acidity		0.6 percent acidity	
		Dipper ratios cream:solution	No. dippers to 1 dipper cream	Dipper ratios cream:solution	No. dippers to 1 dipper cream	Dipper ratios cream:solution	No. dippers to 1 dipper cream
Lime water*	Saturated sol'n. in contact with undissolved $Ca(OH)_2$	1:0.667	1	1:1.241	1	1:1.706	1
Sodium hydroxide	21.5 cc. N/1 per quart	1:1	1	1:1	2	1:1	3
Sodium carbonate	2.3 gm. per quart	1:1	1	1:1	2	1:1	3
Tri-sodium phosphate	7.4 gm. per quart	1:1	1	1:1	2	1:1	3
Farrington solution	64 tablets per quart	1:1	1	1:1.862	1	1:2.713	1

*The odd dipper sizes are necessary here since the dipper size has been adjusted to the solution concentration. The fact that the solution dipper sizes are not as 1:2:3 may arise from the fact that the rule that the percent excess of lime (20 percent) required when cream is neutralized may not be an exactly constant quantity over large ranges of acidity.

of solutions for rapid acid tests at 0.2, 0.4 and 0.6 percent acidities. The 0.4 percent point was studied, because it is considered that with the progress in cream grading that will result from our present grading law, the 0.4 grade will no doubt receive consideration within a few years.

D. **Variation in quart bottle sizes.** In checking the milk bottle volumes, the bottles were filled within 1/8 inch of the offset on which the bottom of the cap rests. This permitted capping the bottles with an ordinary cap or a rubber stopper that fit the aperture designed for the cap. When rubber stoppers were used, especially when the bottles were nearly full, it was necessary to insert a pocket knife blade between the stopper and the bottle while the stopper was inserted. This relieved the air pressure below the stopper and prevented "popping out" of the stoppers.

The volumes of 75 bottles were measured. The average volume was approximately 943 cc., the minimum deviation from the average volume represented an error of 0.5 percent and the maximum error was 1.0 percent. It is considered, therefore, that since all errors were within 0.5 and 1.0 percent, the quart milk bottle is sufficiently reliable to be used in the preparation of rapid acid test solutions.

E. **Errors introduced by variable moisture content of dry reagents weighed.** The chemicals involved here were anhydrous sodium carbonate and trisodium phosphate. With each chemical the moistures were run on freshly opened bottles and on cork-stoppered bottles having been in use in the laboratory for 2 years. Table 5 presents the variations obtained.

These results show that the moisture errors with sodium car-

TABLE 5. ERRORS CAUSED BY VARIATION IN MOISTURE CONTENT OF CHEMICALS WEIGHED.

Chemical	Condition of chemical	Percentage moisture	Difference fresh — old	Weight of reagent per qt. represented by moisture differences
Sodium carbonate	Freshly opened	1.84	0.36%	0.0086 gm. reagent
	Used during 2 yrs.; in cork stoppered bottle	2.20		
Trisodium phosphate	Freshly opened	50.19	1.07%	0.1110 gm. reagent
	used during 2 yrs.; in cork stoppered bottle	51.69		

bonate are not important while those with sodium phosphate are. The sensibility of the moisture balance is 30 mg. The errors introduced by sodium carbonate are but one-fourth the rated sensibility of the balance, while those with the phosphate are nearly four times the rated sensibility. It was decided, therefore, that since this factor cannot be controlled by the operator, phosphate should not be recommended.

F. **Errors introduced through exposure of test solutions to air.** It was considered necessary to know the magnitude of the errors resulting from the exposure of the test solutions to air, since the tendency of any operator would be to allow the test solutions to stand exposed during the working day. The results of these trials are shown in table 6.

These results were obtained by pipetting 100 cc. of solution into several ½ pint fruit jars with screw tops and rubber gaskets which were tightly stoppered until the beginning of the experiment. When the first titration of each series was run the lids were removed from all the jars of that series. At the time intervals shown other 100 cc. portions were titrated.

This experiment shows that all the solutions have some error introduced by exposure and that unless they are kept stoppered between tests or the solution is quickly used, the error may be appreciable. This is particularly true of the sodium hydroxide solution. It will retain its strength very satisfactorily, however, if kept stoppered between tests.

TABLE 6. THE REDUCTION IN STRENGTH AND PERCENTAGE DECREASE IN CONCENTRATION OF RAPID ACID TEST SOLUTIONS WHEN LEFT EXPOSED TO AIR.

Solution employed	Initial* titration cc. N/10 HCl	Time of exposure						
		2 hours	4 hours	6 hours	8 hours	10 hours	12 hours	14 hours
Sodium hydroxide 21.5 cc.N/1 per quart	21.9cc	0.80cc	1.85cc	3.00cc	3.60cc	4.80cc	5.30cc	5.60cc
		3.65%	8.45%	13.70%	16.45%	21.90%	24.2%	25.55%
Sodium carbonate 2.3 gm anhyd. per quart	22.3cc	0.05cc	0.60cc	0.50cc	0.80cc	1.10cc	1.00cc	1.40cc
		0.22%	2.69%	2.25%	3.59%	4.95%	4.49%	6.28%
Farrington solution 64 tablets per quart	29.8cc	0.20cc	0.60cc	0.50cc	0.70cc	0.80cc	1.20cc	0.70cc
		0.67%	2.02%	1.68%	2.35%	2.68%	4.06%	2.35%

*Per 100 cc. test solution.

The initial titrations in table 6 bear out the contention that the Farrington solution (2 tablets per ounce, ie., 64 tablets per quart) contains more neutralizer than is required to react with the acid present in the cream. They likewise substantiate the explanation advanced for the inability of the operator to grade cream at the 0.4 and 0.6 acidity levels because of the variability of the excess alkali and the change in the ratio of alkali to phenolphthalein as the volume of the test solution employed increases.

APPENDIX

PREPARATION OF SOLUTIONS

1. Farrington solution. Dissolve 64 tablets in approximately 1 pint of acid-free[3] distilled water in a quart bottle. Shake with a rotary motion until the tablets are dissolved. Fill to within ⅛ inch of the cap shelf, cap and mix thoroughly.

2. Anhydrous sodium carbonate solution. On a butter moisture scale weigh 2.3 gm. of anhydrous sodium carbonate. To do this tare a small piece of clean, sized paper on the left hand pan with the tare weight of the balance, having both the 1 gm. and 2 gm. slide weights at zero. Then place the 2 gm. slide weight (lower scale) at the 20 percent mark (2 gm.) and the upper or 1 gm. slide weight at the 3 percent mark (0.3 gm.). Add sodium carbonate until the scale balances exactly. Transfer the sodium carbonate quantitatively to a quart bottle, brushing the last traces from the paper with a small artist's (5 and 10-cent store) brush. Add about a pint of acid-free distilled water and rotate to dissolve the reagent. Place the paper back on the moisture balance as before, set the lower weight at 0 percent and the upper one at 5 percent (½ gram). Add phenolphthalein powder until the scale balances. Transfer this powder to the bottle, shake with a rotary motion until the powder is practically all dissolved, fill to within ⅛ inch of the cap shelf, stopper, invert and shake until all the phenolphthalein is dissolved.

3. Sodium hydroxide solution. Place 21.5 cc. of exactly N/1 sodium hydroxide in a quart bottle. To do this, care must be exercised not to spill any of the solution in making the transfer from the shipping container to the quart bottle. The container should be rinsed three or four times with acid-free distilled water, and all the rinsings tranferred quantitatively to the quart bottle. Fill the bottle half full with distilled water; weigh ½ gm. of phenolphthalein powder as described in 2

[3] Acid-free distilled water is specified since the authors are informed that distilled water from battery stations or garages frequently contains sulfuric acid.

above, rotate the solution until the indicator is nearly dissolved. Fill to within ⅛ inch of the cap shelf with distilled water, stopper, invert and shake until all the phenolphthalein is in solution.

4. Saturated lime. Dissolve a piece of quicklime the size of a small hen's egg in hot tap water in a 1-quart fruit jar. Shake this solution at intervals during 3 days, then allow it to stand overnight to permit the sediment to settle and it is then ready for use. When once made it is necessary to shake the solution at least once every 2 days and again allow it to stand overnight before using or it will lose its strength. When the supply of solution is nearly exhausted, dilute again to 1 quart volume with cold tap water, shake several times during the course of a day or two, allow to stand overnight after the last shaking and it is again ready for use. If hot water is used for this dilution it will take longer for the solution again to reach its correct strength since the slaked lime is less soluble in hot than in cold water and a longer time is necessary to again build up its strength.

5. Phenolphthalein solution for use with lime test. Weigh 1.25 gm. phenolphthalein on a moisture scale (set lower weight at 12.5 percent after having tared paper) and dissolve in 50 cc. of 95 percent alcohol. Add 50 cc. of acid-free distilled water. A small amount will precipitate out. Add N/10 sodium hydroxide until the very faintest pink shows. If this requires more than 1.0 cc. the alcohol is not suitable for use. To dissipate the pink color shake the solution vigorously for a short time.